

Superconductivity of noncentrosymmetric $\text{Na}_{11}\text{Hg}_{52}$ and $\text{A}_{11-x}\text{Hg}_{54+x}$ ($\text{A} = \text{Ca}$ or Sr) compounds

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Noncentrosymmetric crystal structures are of significant interest due to the peculiar physical phenomena they frequently promote. Among mercury-based compounds, only a few noncentrosymmetric materials are known, with the vast majority remaining unexplored. In this work, we examine the family of noncentrosymmetric mercurides $\text{Na}_{11}\text{Hg}_{52}$ and $\text{A}_{11-x}\text{Hg}_{54+x}$ ($\text{A} = \text{Ca}$ or Sr). All three materials are identified as new superconductors. The values of the critical temperatures are fairly modest, ranging from 1.7 K ($\text{Ca}_{11-x}\text{Hg}_{54+x}$) to 3.1 K ($\text{Na}_{11}\text{Hg}_{52}$). We postulate that the superconductivity of noncentrosymmetric $\text{Na}_{11}\text{Hg}_{52}$ and $\text{A}_{11-x}\text{Hg}_{54+x}$ ($\text{A} = \text{Ca}$ or Sr) compounds is likely conventional.

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I. INTRODUCTION

Mercury and superconductivity are forever linked as a result of the discovery of zero resistivity in this metal [1]. It was initially selected due to its high purity and an assumption that it would behave as a simple metal, though we now know that the latter is not completely true [2–5]. Even conventional superconductivity of elemental mercury is not so conventional after all, as has been shown recently [6]. It is natural to expect compounds of mercury to host an abundance of interesting physical properties, as has been shown for both chemically simple [7–16] and complex [17–20] materials. The latter are particularly in need of experimental exploration, given the computational difficulties of studying compounds with structural disorder and a large number of atoms per unit cell.

Previously, investigations of noncentrosymmetric $\text{Eu}_{11-x}\text{Hg}_{54+x}$ ($P\bar{6}$ space group) linked its complex magnetic properties to the polyhedra with different volumes, which are able to accommodate either Eu^{2+} or Eu^{3+} species [21,22]. It is a natural question to examine what happens if the magnetic atom is replaced by a nonmagnetic one—especially given the cage-like architecture of this structure. As expected, based on the size similarity between rare-earth (R) and alkali/alkaline-earth (A) ions, previous studies successfully identified isostructural members of the $\text{A}_{11-x}\text{Hg}_{54+x}$ family with $\text{A} = \text{Ca}$ or Sr [18,23,24]. Due to its complexity, the

crystal structure of $\text{A}_{11-x}\text{Hg}_{54+x}$ has been revisited several times. Originally, many of these compounds were described as $R/\text{AHg}_{3.6}$ (for both rare-earth and alkali/alkaline-earth compounds) [25–27], which was later denoted as $\text{Gd}_{14}\text{Ag}_{51}$ [28]. Consequently, the Ca and Sr analogs were found to form in the $\text{A}_{11-x}\text{Hg}_{54+x}$ structure, which, despite the similarities with $\text{Gd}_{14}\text{Ag}_{51}$, has a unique atomic arrangement. For the Eu analog, a slightly different formula— $\text{Eu}_{10}\text{Hg}_{55}$ —was assumed in the literature based on the composition of the investigated sample [22]. The related $\text{Na}_{11}\text{Hg}_{52}$ phase is described by parameter a that is three times larger and is characterized by a strong deformation of the $\text{A}_{11-x}\text{Hg}_{54+x}$ motif, caused by the absence of some Hg sites [24], as highlighted in Fig. 1.

Noncentrosymmetric superconductors—both with and without time-reversal symmetry breaking—remain of interest due to their ability to host unusual superconducting states, as illustrated by CePt_3Si [29], $\text{R}_4\text{Be}_{33}\text{Pt}_{16}$ [30–32], La_7Ni_3 [33], LaNiC_2 [34], NbTaOs_2 [35], $\text{Re}_{24}\text{Ti}_5$ [36], Re_6Ti [37], CaPtAs [38], and BeAu [39–44]. Our previous work identified noncentrosymmetric $\text{Sr}_{11-x}\text{Hg}_{54+x}$ to be a superconductor [18]. Since for the $\text{Ca}_{11-x}\text{Hg}_{54+x}$ and the related $\text{Na}_{11}\text{Hg}_{52}$ compound the ground states were unknown prior to the start of this work, we carried out a comparative study on all three systems.

II. EXPERIMENTAL METHODS

In addition to the reactivity and moisture sensitivity of the alkali and alkaline-earth elements, experimental work with mercurides is also nontrivial—difficulties arise from high vapor pressure, high chemical reactivity, toxicity, and extreme air sensitivity of the resultant compounds [45–47]. We have previously shown [15,18,19,21,48,49] that these challenges

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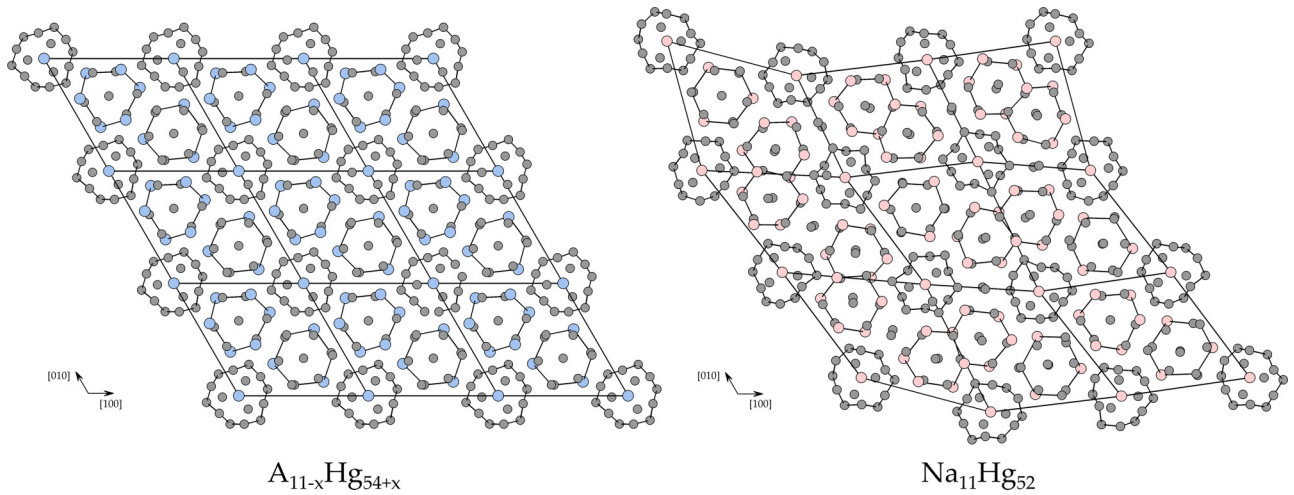


FIG. 1. Visual representation of the differences between the $A_{11-x}Hg_{54+x}$ ($A = \text{Ca}$ or Sr) and $Na_{11}Hg_{52}$ structures. Left: A 3×3 array of the unit cell (solid black lines) for the $A_{11-x}Hg_{54+x}$ structure (blue is A , gray is Hg), adapted from [23]. Right: A single unit cell for the $Na_{11}Hg_{52}$ structure (pink is Na , gray is Hg). The structure has been divided into nine sections, roughly corresponding to the unit cells of $A_{11-x}Hg_{54+x}$ to highlight the differences in the structure. Adapted from refinements of the Na phase synthesized and compared to that from [24].

can be successfully addressed by utilizing a specialized laboratory environment [50].

All samples were synthesized by combining Hg (droplets, Alfa Aesar, 99.999%) with either Na (pieces, Chempur, 99.95%), Ca (pieces, Alfa Aesar, 99.98%), or Sr (pieces, Alfa Aesar, 99.95%). A ratio of A to Hg was varied in order to access the $A_{11-x}Hg_{54+x}$ phase either *via* Hg self-flux or by reacting it stoichiometrically. Samples were sealed in tantalum tubes to prevent evaporation of constituent elements. To protect samples from air and moisture, all syntheses and manipulation were performed in an argon-filled glove box system [50]. The samples were heated up to 500°C and held there for approximately 50 h before cooling slowly over a minimum of 96 h. The samples with excess mercury postsynthesis were placed in specialized crucibles [51] and centrifuged at room temperature to separate crystals from the flux. This method can eliminate most of the mercury; any remainder is removed by placing the crystals on gold foil for several days. Large mm-sized single crystals of the $Ca_{11-x}Hg_{54+x}$ did not have any discernible impurities, while the $Sr_{11-x}Hg_{54+x}$ and $Na_{11}Hg_{52}$ agglomerates have inclusions of secondary phases see Fig. 2. At this stage, we were not able to synthesize the $A_{11-x}Hg_{54+x}$ analogs for $A = \text{Li}, \text{Mg}, \text{K}, \text{Rb}, \text{Cs},$ or Ba .

Powder x-ray diffraction was performed on a Huber G670 Image plate Guinier camera with a Ge-monochromator , located in a glove-box ($\text{CuK}\alpha_1$, $\lambda = 1.54056 \text{ \AA}$). LaB_6 was used as an internal standard for lattice parameter determination. Powders were sealed between two Kapton films to slow down decomposition of the highly reactive powdered material. Phase identification and crystallographic analysis was carried out using WinXPOW [52] and WinCSD software [53]. Lattice parameters were determined by a least-squares refinement using the peak positions, extracted by profile fitting. For full profile fitting of the diffraction pattern by using the Rietveld method, structure models of $\text{Eu}_{11-x}\text{Hg}_{54+x}$ [21] and $\text{Na}_{11}\text{Hg}_{52}$ [24] were used as initial values for the Ca/Sr and Na phases, respectively. XRD data for $\text{Na}_{11}\text{Hg}_{52}$ is shown in the Supplemental Material [54]. Single-crystal diffraction

was not possible due to the extreme air sensitivity of freshly prepared single-crystal samples, coupled with the low melting temperature of these phases, similar to what has been described previously for the Eu analog [21].

Magnetic properties were examined using a Quantum Design (QD) Magnetic Property Measurement System in the temperature range of $1.8\text{--}300 \text{ K}$ and under various applied magnetic fields. Samples were sealed in glass tubes to prevent any reaction with air. Specific-heat data were collected on a QD Physical Property Measurement System from 0.4 to 10 K and under various applied magnetic fields. Due to the high air-sensitivity of all these samples, they were covered with Apiezon N vacuum grease. The corresponding background contribution was subtracted. Due to sample morphology and air-sensitivity, it was not possible to carry out electrical

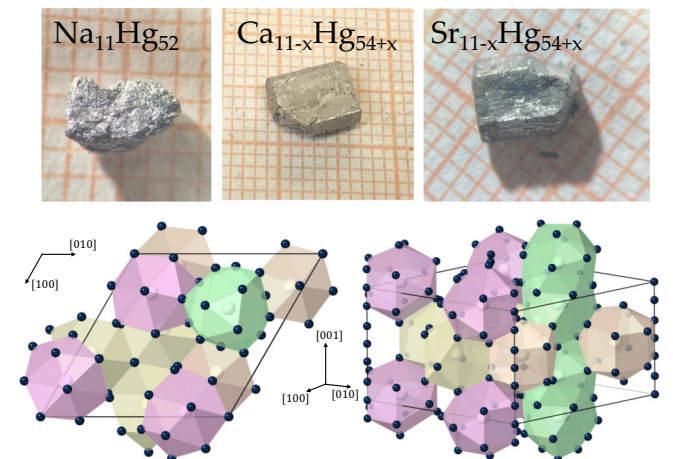


FIG. 2. Top: Samples of $Na_{11}Hg_{52}$, $Ca_{11-x}Hg_{54+x}$, and $Sr_{11-x}Hg_{54+x}$, placed on mm paper. Bottom: The crystal structure of the $A_{11-x}Hg_{54+x}$ ($A = \text{Ca/Sr}$) compounds is cage-like, being composed of four types of A -centered polyhedra with coordination of 14, 15, or 16 [21,55].

TABLE I. Summary of the parameters for the $\text{Na}_{11}\text{Hg}_{52}$ and $\text{A}_{11-x}\text{Hg}_{54+x}$ ($\text{A} = \text{Ca}$ or Sr) phases.

Phase	Lattice parameters ^a (previous work)	Lattice parameters (current work)	T_c (K)	$\mu_0 H_{c,\text{exp}}$ (T)	$\mu_0 H_{c,\text{Pauli}}$ (T)
$\text{Na}_{11}\text{Hg}_{52}$	$a = 39.703(2)$ (13.2343) Å $c = 9.6810(5)$ Å $V = 13215.92$ (1468.44) Å ³ [24]	$a = 39.790(2)$ (13.2633) Å $c = 9.7152(5)$ Å $V = 13321(2)$ (1480.11) Å ³	3.1 ± 0.05	1.0 ± 0.2	5.7
$\text{Ca}_{11-x}\text{Hg}_{54+x}$	$a = 13.389(1)$ Å $c = 9.615(1)$ Å $V = 1492.71$ Å ³ [23]	$a = 13.4365(2)$ Å $c = 9.6564(1)$ Å $V = 1509.80(5)$ Å ³	1.7 ± 0.25	0.2 ± 0.1	3.1
$\text{Sr}_{11-x}\text{Hg}_{54+x}$	$a = 13.602(2)$ Å $c = 9.818(1)$ Å $V = 1573.11$ Å ³ [23]	$a = 13.6339(2) - 13.776(2)$ Å $c = 9.8529(5) - 9.947(2)$ Å $V = 1586.68 - 1634.46$ Å ³	2.2 ± 0.25	0.5 ± 0.1	4.1

^aFor comparison, the $a/3$ and $V/9$ values for $\text{Na}_{11}\text{Hg}_{52}$ are also given.

resistivity measurements, even by utilizing a special resistivity cell [15,48].

III. RESULTS AND DISCUSSION

Motivated by our previous study on strontium mercurides [18], this work investigates superconductivity of the $\text{Na}_{11}\text{Hg}_{52}$ and $\text{A}_{11-x}\text{Hg}_{54+x}$ ($\text{A} = \text{Ca}$ or Sr) noncentrosymmetric compounds. Many mercury-based materials have had their crystal structures extensively examined, but they lack analysis of their physical properties [22–24,56]. The aims of this work were to establish the physical properties of these phases, as well as search for additional members of this family across the alkali and alkaline-earth series. The $\text{Na}_{11}\text{Hg}_{52}$, $\text{Ca}_{11-x}\text{Hg}_{54+x}$, and $\text{Sr}_{11-x}\text{Hg}_{54+x}$ compounds were successfully synthesized and characterized, while the other members of the series still remain out of reach.

Powder x-ray diffraction was used to refine the lattice parameters, which were in agreement with previously reported data—see Table I. The Ca samples were single phase, while some inclusions of secondary phases were found in the Na and Sr samples. For Sr, this has been discussed in depth previously [18]. Both the Ca and Sr compounds are isostructural with the europium mercuride $\text{Eu}_{11-x}\text{Hg}_{54+x}$, investigated by our group recently [21]. However, for the Na system, this structure type could not be refined, and instead a threefold superstructure was obtained. This structure is similar to that of the Ca and Sr systems, albeit with disorder [24,56]. In our study, the structure was confirmed to be $\text{Na}_{11}\text{Hg}_{52}$, in agreement with previous work—the lattice parameters are similar, but differ slightly—see Table I. The corresponding structures are shown in Fig. 1, highlighting the structural difference between $\text{Na}_{11}\text{Hg}_{52}$ and $\text{A}_{11-x}\text{Hg}_{54+x}$ ($\text{A} = \text{Ca}$ or Sr) phases, and Fig. 2, showing an overview of the cage-like structure. Given the difference of the critical temperature values, it is possible that this disorder enhances the T_c of $\text{Na}_{11}\text{Hg}_{52}$, compared to $\text{A}_{11-x}\text{Hg}_{54+x}$ ($\text{A} = \text{Ca}$ or Sr). Further experimental and theoretical studies aimed at understanding the microscopic superconductivity of $\text{Na}_{11}\text{Hg}_{52}$ would therefore be of interest [57–59].

Given the superconductivity of elemental mercury, as well as the high air-sensitivity of the three phases investigated in this work, we have used the methodology previously developed in our group [15,18,19,21,48,49] to extract the intrinsic

properties of $\text{Na}_{11}\text{Hg}_{52}$ and $\text{A}_{11-x}\text{Hg}_{54+x}$ ($\text{A} = \text{Ca}$ or Sr) compounds.

First, we present a detailed analysis of the heat capacity of $\text{Ca}_{10}\text{Hg}_{55}$ [60] in Fig. 3. The temperature dependence of the electronic specific heat C_{el}/T is shown in panel (a) for the temperature range from 0.4 to 2.5 K. The C_{el} was derived by subtracting the phonon contribution from the experimental data, which is given by the βT^3 and δT^5 terms discussed below. A distinct lambda-shaped anomaly indicates a second-order transition, which, in this case, is a superconducting one. An estimate of the Meissner fraction is not possible due to the low value of T_c . The critical temperature was estimated by employing an equal entropy construction, yielding $T_c = 1.74$ K. The low-temperature data were fit by the formula for an s -wave superconductor:

$$\frac{C_{\text{el}}}{T} = \gamma_r + \frac{a}{T} e^{\frac{-\Delta}{k_B T}}, \quad (1)$$

where Δ is the superconducting energy gap and γ_r is the residual linear term, attributed to a small fraction of nonsuperconducting impurity phases. The fit gives $\Delta = 0.23$ meV and

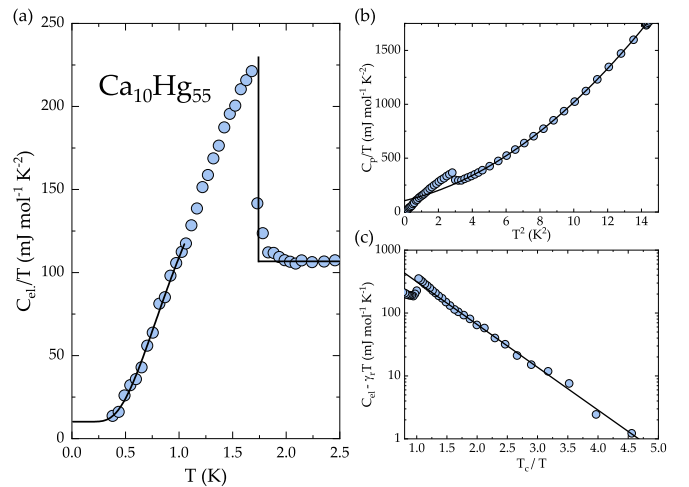


FIG. 3. (a) Low temperature heat capacity of $\text{Ca}_{10}\text{Hg}_{55}$ shows a clear anomaly, associated with the entrance into the superconducting state at $T_c = 1.74$ K. (b) The C_p/T vs. T^2 data. The electronic specific heat C_{el} , reduced by a residual electronic term $\gamma_r T$, vs. reduced temperature T_c/T is shown in panel (c).

$\gamma_r = 10 \text{ mJ mol}^{-1} \text{ K}^{-2}$. The nonzero value of γ_r is consistent with the small amount of impurities, observed in the powder x-ray diffraction data, as discussed above.

In the normal state ($T > T_c$) and at low temperature, C_p/T can be described by the formula

$$\frac{C_p}{T} = \gamma + \beta T^2 + \delta T^4, \quad (2)$$

where γT is the coefficient of the electronic contribution, and βT^3 and δT^5 are the phonon contributions to the specific heat. In Fig. 3(b), C_p/T is plotted as a function of T^2 , with the fit yielding the following parameters: $\gamma = 106 \text{ mJ mol}^{-1} \text{ K}^{-2}$, $\beta = 36 \text{ mJ mol}^{-1} \text{ K}^{-4}$, and $\delta = 5.5 \text{ mJ mol}^{-1} \text{ K}^{-6}$. By subtracting the residual γ_r , the Sommerfeld coefficient for $\text{Ca}_{10}\text{Hg}_{55}$ composition is $\gamma_n = \gamma - \gamma_r = 96 \text{ mJ mol}^{-1} \text{ K}^{-2}$. From γ_n , the normalized specific-heat jump can be calculated, yielding $\Delta C/\gamma_n T_c = 1.35$. This value is close to that expected from BCS theory ($\Delta C/\gamma T_c = 1.43$), indicating that the $\text{Ca}_{10}\text{Hg}_{55}$ system is an *s*-wave, weakly coupled superconductor. It is likely that the $\text{Na}_{11}\text{Hg}_{52}$ and $\text{Sr}_{11-x}\text{Hg}_{54+x}$ superconductors are also conventional. The value of the London penetration depth λ is estimated to be 81 nm, while the coherence length amounts to $\xi = 41 \text{ nm}$. This yields the Ginzburg-Landau parameter $\kappa = 2$, suggesting type II superconductivity. The Debye temperature was calculated from

$$\theta_D = \left(\frac{12\pi^4}{5\beta} nR \right)^{1/3}, \quad (3)$$

where $n = 65$ is the number of atoms per unit cell, and R is the gas constant. The Debye temperature is estimated to be $\theta_D = 152 \text{ K}$. Having θ_D and T_c , the electron-phonon coupling constant can be calculated from the McMillan equation [61]:

$$\lambda_{\text{ep}} = \frac{1.04 + \mu^* \ln\left(\frac{\theta_D}{1.45T_c}\right)}{(1 - 0.62\mu^*) \ln\left(\frac{\theta_D}{1.45T_c}\right) - 1.04}, \quad (4)$$

where the parameter μ^* is a Coulomb repulsion constant. Using a typical value $\mu^* = 0.13$, we obtained the electron-phonon coupling constant for $\text{Ca}_{10}\text{Hg}_{55}$ to be $\lambda_{\text{ep}} = 0.58$, again indicative of a weak-coupling superconductor.

Finally, Figure 3(c) shows the electronic specific heat C_{el} , reduced by a residual electronic term $\gamma_r T$, as a function of reduced temperature T_c/T (on a semilog scale). Linear behavior confirms the assumption that superconductivity in $\text{Ca}_{10}\text{Hg}_{55}$ is of the *s*-wave type since $C_{\text{el}} \sim e^{(-\Delta/k_B T)}$. According to the BCS theory, the energy gap for a superconductor (at $T = 0 \text{ K}$) is given by $\Delta(0) = 3.52k_B T_c$. Hence, taking $T_c = 1.74$ and the expected $\Delta(0) = 0.25 \text{ meV}$, the fit gives a slightly lower $\Delta_{\text{exp}} = 0.23 \text{ meV}$ value.

For the $\text{Na}_{11}\text{Hg}_{52}$ and $\text{Sr}_{11-x}\text{Hg}_{54+x}$ compounds, a similar analysis of specific-heat data was not possible, as a result of the small heat capacity signal, coupled with large background contribution (as a result of large amount of grease needed to protect the samples from decomposition). Magnetic susceptibility data for $\text{Na}_{11}\text{Hg}_{52}$ and $\text{Sr}_{11-x}\text{Hg}_{54+x}$ are shown in the Supplemental Material [54]. The corresponding values of T_c and H_c are summarized in Fig. 4 for all three compounds. The

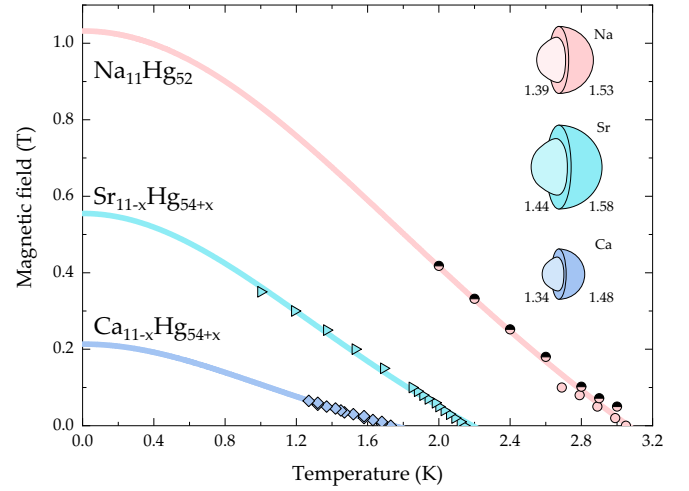


FIG. 4. The H - T phase diagram for the $\text{Na}_{11}\text{Hg}_{52}$ (pink) and $\text{A}_{11-x}\text{Hg}_{54+x}$ [$A = \text{Ca}$ (blue) or Sr (cyan)] compounds. Inset: the ionic and atomic radii of Na, Ca, and Sr elements are shown.

fit to the Ginzburg-Landau relation

$$\mu_0 H_c(T) = \mu_0 H_c(0) \frac{1 - \left(\frac{T}{T_c}\right)^2}{1 + \left(\frac{T}{T_c}\right)^2} \quad (5)$$

is shown as a solid line. All of the critical field values, extracted from these fits, are substantially larger than elemental mercury ($\mu_0 H_c = 0.03 \text{ T}$), as well as other conventional mercury-based superconductors (for example, $\text{LaHg}_{6.4}$ with $\mu_0 H_{c2} = 0.06 \text{ T}$ [48] or Lu/YHg_3 with $\mu_0 H_{c2} = 0.08 \text{ T}$ [15]). However, for all three compounds the H_c values are below the Pauli limiting fields—see Table I. Furthermore, as discussed previously for the $\text{Sr}_{11-x}\text{Hg}_{54+x}$ compound [18], the superconductivity is robust against structural disorder—the mixed occupancy on one of the sites (A4 [21]) changes the lattice parameters significantly, while the value of T_c remains fairly unaffected. These characteristics further support conventional superconductivity of these mercurides. Due to their noncentrosymmetric structures, muon spin relaxation, rotation, and resonance as well as other investigations of $\text{Na}_{11}\text{Hg}_{52}$ and $\text{A}_{11-x}\text{Hg}_{54+x}$ ($A = \text{Ca}$ or Sr) compounds could be of interest [62–66].

IV. CONCLUSION

Our earlier work identified $\text{Sr}_{11-x}\text{Hg}_{54+x}$ as a new superconductor, which inspired a question regarding possible superconductivity of structurally similar mercurides based on alkali and alkaline-earth elements. In this study, superconductivity was confirmed for the $\text{Ca}_{11-x}\text{Hg}_{54+x}$ analog and related $\text{Na}_{11}\text{Hg}_{52}$ phase, while the synthesis of the Li, Mg, K, Rb, Cs, and Ba systems was not successful.

The $\text{Na}_{11}\text{Hg}_{52}$ and $\text{A}_{11-x}\text{Hg}_{54+x}$ ($A = \text{Sr}$ or Ca) noncentrosymmetric compounds display conventional superconductivity below $T_c = 3.1 \pm 0.05 \text{ K}$ ($\text{Na}_{11}\text{Hg}_{52}$), $T_c = 1.7 \pm 0.25 \text{ K}$ ($\text{Ca}_{11-x}\text{Hg}_{54+x}$), and $T_c = 2.2 \pm 0.25 \text{ K}$ ($\text{Sr}_{11-x}\text{Hg}_{54+x}$). While their critical fields are significantly larger than that of elemental mercury ($0.2 \text{ T} \leq H_{c2} \leq 1 \text{ T}$), these compounds most likely host conventional superconductivity, as indicated

by the strength of electron-phonon coupling, as well as the size of the jump in the specific-heat data.

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DATA AVAILABILITY

The data that support the findings of this article are not publicly available upon publication because it is not technically feasible and/or the cost of preparing, depositing, and hosting the data would be prohibitive within the terms of this research project. The data are available from the authors upon reasonable request.

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